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# Use of Malathion-Treated Drying Trays To Protect RAISINS From Insects During Drying and Storage

Marketing Research Report No. 789

Agricultural Research Service  
UNITED STATES DEPARTMENT OF AGRICULTURE

## PREFACE

This publication is based on a large-scale study of the use of malathion-treated raisin-drying trays as a means of protecting raisins from insects during the periods of drying and storage. The tests also included laboratory bioassay tests and microanalyses. A number of individuals and groups cooperated in this study. Preston L. Hartsell, Agricultural Research Service, Stored-Product Insects Research Branch, Dried Fruit and Tree Nut Insects Investigations, at Fresno, Calif., assisted in this study by drawing samples, cleaning and processing them, and making insect counts, bioassay determinations, and moisture readings. The Stored-Product Insects Research and Development Laboratory, at Savannah, Ga., performed the malathion residue determinations on the grapes, trays, and raisins.

The malathion was applied to the paper trays under the direction of K. G. Hadley, Associate Chemist, Packaging Grades Development, Crown Zellerbach Corporation, Central Research Division, Camas, Wash.

Vineyardists Ed Steffen, of Biola, and Harper Gabrielson, of the Lone Star area near Fresno, Calif., dried the raisins and provided space for storage.

The California Raisin Advisory Board assisted in obtaining the cooperating vineyardists and the raisins used in the study. The Department of Viticulture, Fresno State College, cooperated by providing the raisin-processing equipment. Professor V. E. Petrucci and Nick Dokoozlian of the college supervised the processing of the raisins.

The U.S. Department of Agriculture, Consumer and Marketing Service, Fruit and Vegetable Division, Processed Products Standardization and Inspection Branch, Fresno, Calif., conducted microanalysis determinations on the natural and processed raisins.

The Dried Fruit Association of California also made microanalysis determinations and arranged for the taste panel tests.

Dr. Martin A. Miller, Department of Food Science and Technology, University of California, Davis, Calif., performed the taste panel studies.



## PRECAUTIONS

Insecticides used improperly can be injurious to man and animals. Use them only when needed and handle them with care. Follow the directions and heed all precautions on the labels.

Some States have special restrictions on the use of certain insecticides. Before applying insecticides, check State and local regulations.

Keep insecticides in closed, well-labeled containers in a dry place. Store them where they will not contaminate food or feed, and where children and animals cannot reach them. Promptly dispose of empty insecticide containers; do not use for any other purpose.

# Use of Malathion-Treated Drying Trays To Protect RAISINS From Insects During Drying and Storage

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## SUMMARY

In tests of malathion as a protectant for raisins against insect infestation during drying and storage, paper trays were impregnated with malathion and grapes were dried into raisins on them. Most of the results of the 1961-62 tests have been reported elsewhere (2).<sup>1</sup> The complete results of the 1962-63 tests are presented here in three parts: Evaluation of malathion residues and insect populations on the trays and raisins in the field and in storage; laboratory bioassay of raisins; and microanalysis of the raisins. The data on microanalysis of the raisins in the 1961-62 tests has not previously been published.

*Field Evaluation, Including Storage*—Thompson Seedless grapes that had been dusted on the vine with a 4-percent malathion dust contained less than 0.5 parts per million (p.p.m.) of malathion 3 days later when picked. Dusted grapes were placed on paper drying trays that contained malathion residues of about 30, 150, and 300 milligrams per square foot (mg./sq.ft.). Of the malathion on the trays, 86.6 to 96.5 percent was lost during drying of the raisins. The raisins dried on these trays picked up from 0.9 to 9.5 p.p.m. of malathion. In most instances, one year of storage reduced the amount of malathion on the raisins, and processing also removed some of it. A malathion residue of 9.4 p.p.m. did not affect the odor or flavor of the raisins.

At first raisin moths predominated on the trays; then dermestids, Indian-meal moths, and saw-toothed grain beetles became more numerous during storage. At least 44 different species of insects and mites were among the raisins during the drying period. Nearly all insects on the

treated trays were dead. At the end of storage, significantly fewer live insects were found among raisins dried on treated trays than among those dried on untreated trays.

*Laboratory Bioassays*—Samples of natural and processed raisins were exposed to adults of Indian-meal moths and saw-toothed grain beetles in the laboratory at the end of the drying period in the vineyards and again after 3, 6, 9, and 12 months of outdoor storage. Malathion at 2 p.p.m. or more on the raisins virtually prevented insect development and reproduction for at least one year. Removal of some of the malathion by processing allowed an increase of insect activity. Processing increased the moisture content, but after 3 or 4 months in the laboratory, the moisture contents of natural and processed raisins were the same. The moisture content increased during the first 6 months the raisins were held in outdoor storage.

*Microanalyses*—As the 1961-62 storage began, raisins dried on malathion-treated trays generally contained more dead insects and insect fragments than raisins dried on untreated trays, but after 12 months in storage, raisins from untreated trays had more insect fragments. Processing of the natural raisins greatly reduced the amount of foreign material.

The 1962-63 studies showed no difference in the amount of insect material in processed raisins between those from treated and untreated trays. Putting natural raisins through a raisin cleaner at the time of harvest removed at least 90 percent of the insects and reduced the amount of foreign material on the raisins.

<sup>1</sup> Italic numbers in parentheses refer to Literature Cited, p. 14.

## INTRODUCTION

During the summer of 1961, preliminary and large-scale field tests were begun to determine the effectiveness of malathion-treated paper raisin-drying trays in protecting raisins from in-

sects during drying and storage. The results of that study indicated that malathion-treated trays were effective in protecting the raisins from insect attack during the drying period, and

that sufficient malathion migrated to the raisins to protect them from insects for 1 year in farm-type storage. The results of the preliminary studies and the first 5 months of the large-scale field tests were so promising that a commercial-type study was begun during the summer of 1962 and continued into the fall of 1963 (2).

In order for us to conduct these experiments with malathion in 1962 and 1963, a temporary exemption from the requirement for a food additive tolerance was issued by the Food and Drug Administration to treat 20 tons of raisins. Included as a condition of granting this temporary exemption was the requirement that all raisins be destroyed that had a residue of malathion in excess of 5 parts per million as a result of drying the raisins on paper treated with mala-

thion. On the basis of other data and results reported in this study, the FDA later established a permanent tolerance of 8 p.p.m. on ready-to-eat raisins. The only condition was that the level of malathion deposit on the trays on which such raisins were dried would not exceed 200 mg./sq.ft.

The results of the 1962-63 study are reported in three parts. In part I, the levels of malathion residue and the insect populations were determined and taste tests were made. In part II, bioassay studies evaluated the effectiveness of the different levels of malathion residue under controlled laboratory conditions. In Part III, insect and insect fragment counts were determined by a laboratory washing process (microanalysis).

## PART I—FIELD EVALUATION, INCLUDING STORAGE

This part is concerned with the malathion residues on the trays and on the raisins in the 1962-63 studies and with the insect populations in these raisins in the field and in storage.

### Materials and Methods

#### Treatment of Trays

The raisin trays used in this study were the standard paper trays, 2 feet wide and 3 feet long. An emulsifiable concentrate of premium-grade 57-percent malathion was diluted with water and applied to the paper of the trays during the final phase of the paper manufacturing. Four lots of trays were prepared. One lot contained untreated trays, and it was desired to have malathion deposits of 100, 200, and 400 mg./sq.ft. on the remaining three lots. However, the amount deposited by the applicator machine was not entirely predictable, and the actual deposits measured about 30, 150, and 300 mg./sq.ft.

#### Treatment of Grapes

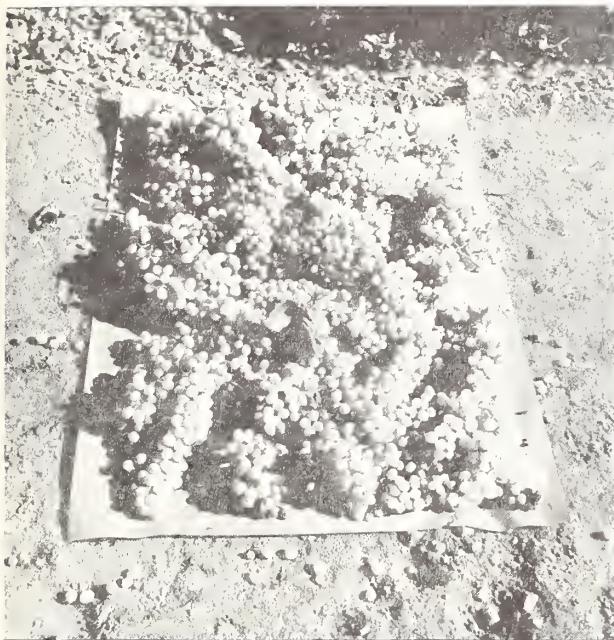
About 5 acres of Thompson Seedless grapes in each of two vineyards in the Fresno area were used in this study. About one-fifth of the vines in each of the 5-acre plots were dusted with a premium-grade 4-percent malathion dust applied at the rate of 35 lb./acre. The dust was applied with standard field-dusting equipment. The grapes were removed from the dusted vines 3 days after treatment and placed on paper trays to dry. Since numerous vineyardists use malathion dust as a means of controlling insects that attack the grapevines, it was important to know the amount of malathion remaining on the grapes at the time they were placed on the trays.

#### Picking and Drying of Grapes

The 5 acres of grapes in each of the two vineyards were picked, placed on trays, turned, rolled, collected from the trays, and placed in sweat boxes by commercial crews. These boxes normally hold 180 lb. of raisins per box. The term "sweat" comes from the conditioning of the raisins; that is, an equalization of moisture content.

Vineyard No. 1, located in the Lone Star area about 7 miles east of Fresno, consisted of young, vigorous vines. The grapevines on one acre were dusted. Grapes were removed from these vines and placed on malathion-treated paper trays on August 30, 1962 (fig. 1). About half of the trays had been treated with malathion at the rate of 152.4 mg./sq.ft., and the other half at the rate of 304.2 mg./sq.ft. Grapes from the 4 acres of undusted vines were picked and placed on malathion-treated trays and on control, or untreated, paper trays on August 28 and 29, 1962. Grapes from one acre were placed on each of the following types of trays: Untreated, 31.2, 152.4, and 304.2 mg./sq.ft. All of the trays were turned on September 7 and 8 and were rolled into biscuit rolls (fig. 2) on September 13, 14, and 15. The raisins were removed to sweat boxes and placed in farm-type storage on September 28, 29, and 30, 1962.

Vineyard No. 2, located in the Biola area about 12 miles west of Fresno, contained more mature vines than vineyard No. 1. The dust was applied to 1 acre of grapevines on September 2, 1962, and the grapes were picked 3 days later. They were placed on trays treated with malathion at the rate of 150.2 and 290.4 mg./sq.ft. On September 5, 6, and 7 the 4 acres of undusted grapes



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FIGURE 1.—Fresh grapes placed on paper trays to dry.



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FIGURE 2.—Some of the trays were rolled into "biscuit rolls" to complete the raisin-drying process.

were picked and put on untreated trays, and trays treated at 34.2, 150.2, and 290.4 mg./sq.ft. On September 21, the drying grapes were turned on the trays, and on September 23, the trays were rolled into cigarette rolls (fig. 3). The raisins were removed from the trays, dumped into sweat boxes, and placed in farm-type storage on October 1. Both biscuit and cigarette rolls are used by the raisin industry, and it just happened that in this study one of the vineyardists used biscuit and the other cigarette rolls.

#### *Storage of Raisins*

The sweat boxes containing raisins dried on trays treated with a given dosage of malathion were placed together as a unit in the stack.

In vineyard No. 1, the stack was 1 box wide, 7 boxes high, and 12 boxes long. In vineyard No. 2, it was 1 box wide, 10 boxes high, and 8 boxes long. The bottom boxes rested on 4- by 4-in. planks. New sweat boxes were used. When stacked, these boxes fitted tightly enough together to exclude rodents, but would permit insects to enter. The surface of the top row of boxes was covered with  $\frac{1}{4}$ -in. mesh hardware cloth as a bird and rodent barrier. A dismountable, pitched roof frame was constructed on top of each stack and covered with roofing paper to keep out the rain. Wooden raisin-drying trays were tacked to the sides and ends of both stacks for additional protection from weather (fig. 4).

#### *Malathion Residue on Samples*

Malathion residues were determined on the grapes when they were placed on the trays, and on the trays at the beginning and end of the drying period in the vineyard. Residue analyses were also made on natural and processed raisins at the end of the field-drying period, and at intervals of 3, 6, 9, and 12 months during farm-type storage.

Two 1-quart samples of dusted grapes were removed from the vines 3 days after the dusting of the vines. The samples were taken at random from bunches of grapes in the dusted area and placed directly in lacquered metal cans. The cans were promptly sealed; and the samples were quick-frozen, packed in dry ice, and shipped by air express to the analytical laboratory where the residue analyses were made. Samples of undusted grapes were submitted in the same way.

At the time the trays were placed in each vineyard, five trays were taken at random from each tray treatment, packaged in separate plastic containers, and sent by airmail express to the analytical laboratory. The same procedure was used in sampling trays from each vineyard at the end of the drying period in the field.

When the raisins were removed from the trays and placed in sweat boxes to be stored, two sweat boxes were set aside from each tray-treatment combination in each vineyard. These two sweat boxes from each treatment were mixed together



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FIGURE 3.—Trays were also rolled into "cigarette" rolls to complete the raisin-drying process.



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FIGURE 4.—Type of farm storage in which the raisins were held for 12 months.

and returned to their original boxes, and three 1-quart samples of natural raisins were taken for residue analyses. Each sample was placed in a lacquered tin can, sealed, and forwarded for residue analysis. This procedure was followed for each stack at the end of 3, 6, 9, and 12 months in storage.

#### *Processing of Raisins*

A pilot-scale processing unit, which had all of the important features of the larger industry units of the raisin industry, was used to process the raisins. The raisins were sorted, screened, washed, inspected, and packed in cartons.

#### *Insect Counts*

At the time the raisins were removed from the trays and placed in storage, screenings from 10 trays from each treatment in each vineyard were examined for the presence and abundance of insects. At the end of 3, 6, 9, and 12 months in storage, screenings from two sweat boxes of raisins were examined from each treatment from each storage, except that no samples were taken of the dusted grapes dried on trays treated at 150.2 and 290.4 mg./sq.ft. One sweat box of raisins dried from dusted grapes was examined at each interval from each tray treatment for those stored near vineyard No. 1.

#### *Taste Panel Tests*

Two 1-quart samples of processed raisins containing no malathion and two 1-quart samples of processed raisins containing 9.4 p.p.m. of malathion were subjected to taste evaluation tests.

## Results

#### *Malathion Residue on Trays*

At the time the paper raisin-drying trays were placed in the vineyards, the approximate amounts of malathion on the trays were 30, 150, and 300 mg./sq.ft. This was from  $\frac{1}{4}$  to  $\frac{2}{3}$  less than originally planned for this test. The greatest loss of malathion from the trays occurred during the drying period. From 86.6 to 96.5 percent of the malathion was lost during that time (table 1).

#### *Malathion Residue on Grapes*

Three days before the grapes were to be picked for drying into raisins, a 4-percent malathion dust was applied at the rate of 35 lb. per acre, or 1.4 lb. of actual insecticide per acre. Malathion residues were determined on samples of these grapes removed from the vine. Less than 0.5 p.p.m. of malathion remained on the grapes at the time they were picked.

#### *Malathion Residue on Raisins*

Raisins dried on malathion-treated trays picked up malathion during the drying period. The raisins dried in vineyard No. 1 picked up more malathion from the trays than those dried in vineyard No. 2. There was some indication that dusted grapes produced raisins with higher malathion deposits. In most instances there was less malathion on the raisins at the end of 12 months than at the beginning of storage, but this loss was small. Processing the raisins did remove some of the malathion (table 2).

TABLE 1.—Average malathion residues<sup>1</sup> on paper raisin-drying trays at the beginning and end of drying period

Vineyard	Beginning of drying period	End of drying period	Loss of original deposit
	Mg./sq. ft.	Mg./sq. ft.	Percent
No. 1	31.2	2.1	93.3
No. 1	152.4	20.4	86.6
No. 1	304.2	14.6	95.2
No. 1 <sup>2</sup>	< 0.5	< 0.5	—
No. 2	34.2	1.2	96.5
No. 2	150.2	12.5	91.7
No. 2	290.4	14.2	95.1
No. 2 <sup>2</sup>	< 0.5	< 0.5	—

<sup>1</sup> Average residue from 5 trays. The sensitivity of the chemical analytical method is 0.5 p.p.m.

<sup>2</sup> Untreated check.

### Insects in Raisin Screenings

Examination of raisin screenings at the end of the drying period revealed that at least 44 different species of insects and mites traversed the trays while the grapes were drying in the vineyards. The number of dead insects found on the treated trays indicated that the malathion

residues afforded the raisins some protection. Insects recovered from screenings of natural raisins from the two stacks at the end of 3, 6, 9, and 12 months in storage indicated that the live insect population was relatively small in both stacks in comparison to the number of live insects found among the screenings during the 1961 studies (table 3). Fewer live insects were observed among the raisins stored near vineyard No. 1 than among those near vineyard No. 2. This was apparently due to the greater amount of malathion present on the raisins dried in vineyard No. 1.

During the first 9 months of storage, raisin moths were the most numerous among the insect population. During the last 3 months in storage, dermestids, Indian-meal moths, and saw-toothed grain beetles predominated. In both vineyards, at the end of 12 months in farm-type storage, 99.7 and 95 percent fewer live insects were found in the screenings from raisins dried on the trays with the higher rates of malathion treatment than those dried on untreated trays.

### Taste Panel Tests

A malathion residue of 9.4 p.p.m. did not affect the odor or flavor of processed raisins.

TABLE 2.—Average malathion residues<sup>1</sup> found on natural and processed raisins during 1 year in storage

[Undusted grapes and grapes dusted with malathion were placed on untreated and malathion-treated trays to dry.]

Vineyard, treatment of grapes on vines, and residue on trays	Natural raisins					Processed raisins				
	At beginning of storage	At end of—				At beginning of storage	At end of—			
		3 months	6 months	9 months	12 months		3 months	6 months	9 months	12 months
<b>Vineyard No. 1</b>	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m.	P.p.m.
Undusted grapes:										
Untreated trays.....	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
31.2 mg./sq. ft.....	1.3	1.7	1.6	1.1	0.8	0.9	1.3	1.4	1.2	0.7
152.4 mg./sq. ft.....	4.8	6.3	5.6	3.6	3.3	4.7	4.8	4.0	3.4	3.5
304.2 mg./sq. ft.....	7.7	15.3	10.0	8.4	9.4	7.5	7.4	6.8	5.4	6.1
Dusted grapes: <sup>2</sup>										
152.4 mg./sq. ft.....	4.6	5.5	6.5	5.2	4.7	3.9	4.3	4.3	2.3	3.8
304.2 mg./sq. ft.....	9.5	13.0	9.6	9.4	8.9	9.6	8.6	7.1	6.0	5.7
<b>Vineyard No. 2</b>										
Undusted grapes:										
Untreated trays.....	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
34.2 mg./sq. ft.....	0.9	0.7	0.7	< 0.5	0.6	0.6	< 0.5	< 0.5	< 0.5	< 0.5
150.2 mg./sq. ft.....	2.1	2.3	1.9	1.5	1.4	1.5	1.5	1.2	1.3	1.1
290.4 mg./sq. ft.....	3.7	3.4	3.1	3.3	2.6	2.5	2.3	2.0	2.4	1.9
Dusted grapes: <sup>2,3</sup>										
150.2 mg./sq. ft.....	2.4	—	—	—	—	—	—	—	—	—
290.4 mg./sq. ft.....	4.7	—	—	—	—	—	—	—	—	—

<sup>1</sup> The sensitivity of the chemical analytical method is 0.5 p.p.m. Residues are averages of 3 replications.

<sup>2</sup> Treated with 4% malathion.

<sup>3</sup> The commercial crew harvested part of these raisins by mistake, and there were not enough for residual studies.

TABLE 3.—Number of insects found in screenings removed from natural raisins during 1 year in storage<sup>1</sup>

[Undusted grapes and grapes dusted with malathion were placed on untreated and malathion-treated trays to dry.]

Vineyard, treatment of grapes, and malathion residue <sup>2</sup> on trays	At beginning of storage		At end of 3 months		Live insects at end of <sup>3</sup>		
	Live	Dead	Live	Dead	6 months	9 months	12 months
Vineyard No. 1	Insects	Insects	Insects	Insects	Insects	Insects	Insects
Undusted grapes:							
Untreated trays	96	2	260	52	69	52	1,652
31.2 mg./sq. ft.	3	49	0	398	0	4	432
152.4 mg./sq. ft.	0	104	0	352	0	0	60
304.2 mg./sq. ft.	0	174	8	440	1	0	4
Dusted grapes: <sup>4</sup>							
152.4 mg./sq. ft.	1	347	0	192	0	0	0
304.2 mg./sq. ft.	0	34	0	264	0	0	0
Vineyard No. 2							
Undusted grapes:							
Untreated trays	103	77	104	192	109	270	5,217
34.2 mg./sq. ft.	7	219	264	309	5	66	408
150.2 mg./sq. ft.	1	377	32	280	0	26	304
290.4 mg./sq. ft.	2	307	8	384	4	8	264
Dusted grapes: <sup>4,5</sup>							
150.2 mg./sq. ft.	0	399	—	—	—	—	—
290.4 mg./sq. ft.	0	350	—	—	—	—	—

<sup>1</sup> Counts at beginning were based on screenings from 10 trays from each tray and grape treatment combination. Counts at end of 3, 6, 9, and 12 months were based on screenings from two sweat boxes of natural raisins from each tray and grape combination.

<sup>2</sup> The sensitivity of the chemical analytical method is 0.5 p.p.m.

<sup>3</sup> Dead insects were not counted in these samples.

<sup>4</sup> Treated with 4% malathion.

<sup>5</sup> The commercial crew harvested part of these raisins by mistake, and not enough were available for the entire storage period.

## PART II—LABORATORY BIOASSAYS

This part describes the bioassay tests that were made with the raisins in the 1962–63 studies, both before and after processing.

### Materials and Methods

#### Insects

Saw-toothed grain beetles, *Oryzaephilus surinamensis* (L.) and Indian-meal moths, *Plodia interpunctella* (Hübner), were used as the test insects for these bioassays. The moths were reared on a medium containing honey, glycerol, and poultry mash. The beetles were reared on rolled oats and brewer's yeast. The rearing laboratory was kept at a constant temperature of  $80^{\circ} \pm 1^{\circ}$  F. and R.H. of  $50 \pm 2$  percent, except during the period March through August, when it varied from about  $70^{\circ}$  to  $80^{\circ}$  F. The laboratory was moved during this period, and thermostatic control was not possible.

#### Raisin Samples

At the end of the drying period in the vineyards, the natural raisins were placed in protected outdoor storage. At this time, two boxes of raisins from each treatment level were set

aside in the laboratory. Raisins from one box of each treatment level were processed, and raisins from the other box were left as natural raisins.

From each box of raisins that had been set aside for testing, ten  $\frac{1}{2}$ -lb. samples were taken. Each  $\frac{1}{2}$ -lb. sample was placed in a widemouthed mason jar. The top was closed with filter paper, held in place by a metal ring screwed onto the jar.

About 100 adult saw-toothed grain beetles were placed in each of five sample jars from each box. About 35 adult Indian-meal moths were placed in the other five jars from each box of raisins.

At the end of 30 days all samples were examined to determine if the insects had been able to survive and produce progeny on the raisins. Those samples containing saw-toothed grain beetles were shaken on a No. 10 standard Taylor<sup>2</sup> screen, which removed the insects from the rai-

<sup>2</sup> Trade names are used in this publication solely for the purpose of providing specific information. Mention of a trade name does not constitute a guarantee or warranty of the product by the U.S. Department of Agriculture or an endorsement by the Department over other products not mentioned.

sins. Samples containing Indian-meal moths were emptied into a flat pan and examined. Those samples containing fewer than five live insects were returned to the original jar and re-infested; those containing five or more live insects were removed from the test.

Other samples of raisins were taken from the same boxes at the end of 3, 6, and 9 months in storage. Insects were added to these samples in the same manner as to the first ones. The samples were examined for insects three times at monthly intervals. The 12-month samples were held only 1 month for examination.

The moisture content of the raisins was determined at the time the samples were taken and at monthly intervals during 1 year of storage. This was done with a dried-fruit moisture tester developed by the Dried Fruit Association of California.

## Results

The amount of malathion found and the number of insects living in the samples of raisins that were subjected to the conditions of commercial storage up to 12 months before exposure to insect infestations in jar tests in the laboratory are shown in tables 4 and 5.

TABLE 4.—*Malathion residue on trays and on natural and processed raisins sampled during 1 year in farm-type storage at vineyard No. 1, and number of insects alive in samples 1 month after infestation of samples in the laboratory*

Malathion residue on trays and time in storage	Natural raisins			Processed raisins		
	Malathion residue <sup>1</sup>	Live insects <sup>2</sup>		Malathion residue <sup>1</sup>	Live insects <sup>2</sup>	
		Indian-meal moth	Saw-toothed grain beetle		Indian-meal moth	Saw-toothed grain beetle
< 0.5 mg./sq. ft.:	P.p.m.	Number	Number	P.p.m.	Number	Number
Before storage.....	< 0.5	30	185	< 0.5	13	171
3 months.....	< 0.5	182	176	< 0.5	168	190
6 months.....	< 0.5	80	205	< 0.5	117	221
9 months.....	< 0.5	220	162	< 0.5	150	154
12 months.....	< 0.5	58	177	< 0.5	48	255
31.2 mg./sq. ft.:						
Before storage.....	1.3	0	0	0.9	0	13
3 months.....	1.7	0	0	1.3	6	12
6 months.....	1.6	0	0	1.4	3	1
9 months.....	1.1	1	0	1.2	15	5
12 months.....	0.8	1	0	0.7	8	7
152.4 mg./sq. ft.:						
Before storage.....	4.8	0	0	4.7	0	0
3 months.....	6.3	0	0	4.8	0	0
6 months.....	5.6	0	0	4.0	0	0
9 months.....	3.6	0	0	3.4	0	0
12 months.....	3.3	0	0	3.5	0	0
304.2 mg./sq. ft.:						
Before storage.....	7.7	0	0	7.5	0	0
3 months.....	15.3	0	0	7.4	0	0
6 months.....	10.0	0	0	6.8	0	0
9 months.....	8.4	0	0	5.4	0	0
12 months.....	9.4	0	0	6.1	0	0

<sup>1</sup>The sensitivity of the chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Average of 5 samples.

The raisins lost some malathion during the storage period, but the loss was not great. Processing the raisins reduced the amount of malathion as well as the length of time the raisins were protected. The Indian-meal moths and saw-toothed grain beetles had difficulty in surviving and developing on raisins that contained a malathion residue of 2 p.p.m. or more.

The raisins dried in vineyard No. 2 contained less malathion than those dried in vineyard No. 1 and were more readily attacked by insects. This was particularly true of the raisins dried on trays with residues of 34 and 150 mg. of malathion per sq.ft. The reason for the variation in malathion residues between the two vineyards is not known. Residues in vineyard No. 1 were very erratic, particularly those resulting from heavier dosages.

Processing increased the moisture content of the raisins (tables 6 and 7). When both natural and processed raisins were held in the laboratory for 1 year after the first processing, the moisture contents were about equal after 5 months. The moisture content of the raisins gradually declined from March through August. This was due to the fact that the laboratory was moved during the period and a source of constant

TABLE 5.—*Malathion residue on trays and on natural and processed raisins sampled during 1 year in farm-type storage at vineyard No. 2, and number of insects alive in samples 1 month after infestation of samples in the laboratory*

Malathion residue on trays and time in storage	Malathion residue <sup>1</sup>	Natural raisins		Malathion residue <sup>1</sup>	Processed raisins		
		Live insects <sup>2</sup>			Live insects <sup>2</sup>		
		Number	Number		Number	Number	
< 0.5 mg./sq. ft.:	P.p.m.	Number	Number	P.p.m.	Number	Number	
Before storage.....	< 0.5	159	246	< 0.5	120	295	
3 months.....	< 0.5	171	187	< 0.5	194	167	
6 months.....	< 0.5	199	227	< 0.5	154	338	
9 months.....	< 0.5	304	155	< 0.5	234	372	
12 months.....	< 0.5	189	204	< 0.5	109	248	
34.2 mg./sq. ft.:							
Before storage.....	0.9	0	0	0.6	11	79	
3 months.....	0.7	3	0	< 0.5	40	95	
6 months.....	0.7	2	2	0.5	53	126	
9 months.....	< 0.5	18	2	< 0.5	166	99	
12 months.....	0.6	9	0	< 0.5	91	87	
150.2 mg./sq. ft.:							
Before storage.....	2.1	0	0	1.5	0	6	
3 months.....	2.3	0	0	1.5	2	2	
6 months.....	1.9	0	0	1.2	0	0	
9 months.....	1.5	1	0	1.3	1	1	
12 months.....	1.4	2	0	1.1	1	1	
290.4 mg./sq. ft.:							
Before storage.....	3.7	0	0	2.5	0	0	
3 months.....	3.4	0	0	2.3	0	0	
6 months.....	3.1	0	0	2.0	0	0	
9 months.....	3.3	0	0	2.4	0	0	
12 months.....	2.6	2	0	1.9	0	0	

<sup>1</sup>The sensitivity of the chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Average of 5 samples.

humidity was not available (table 6). This reduction in moisture apparently had little effect on insect development.

The moisture content of the raisins in farm-

type storage increased during the first 6 months and decreased during the last 6 months. This was probably due to the effect of the rainy season during the first 6 months of the storage period and the warm dry weather during the last 6 months.

TABLE 6.—*Average moisture readings of natural and processed raisins at the time they were placed in the laboratory and at monthly intervals for 1 year*

[The raisins from the 2 vineyards were combined for these analyses.]

Storage in laboratory	Natural raisins	Processed raisins
	Percent	Percent
None.....	13.0	15.7
1 month.....	13.9	15.7
2 months.....	16.0	16.1
3 months.....	14.5	16.7
4 months.....	13.2	14.7
5 months.....	10.3	11.0
6 months.....	10.3	10.7
7 months.....	10.7	11.1
8 months.....	10.5	10.8
9 months.....	10.7	10.7
10 months.....	11.3	11.1
11 months.....	11.3	11.3
12 months.....	12.5	12.5

TABLE 7.—*Moisture content of raisins when they were removed from the two outdoor storages at 3-month intervals during the 1-year storage period*

Vineyard and length of storage on farm	Before processing	After processing
	Percent	Percent
<b>Vineyard No. 1</b>		
Before storage.....	13.7	16.7
3 months.....	14.8	17.8
6 months.....	14.4	16.7
9 months.....	11.6	14.1
12 months.....	10.4	12.3
<b>Vineyard No. 2</b>		
Before storage.....	10.2	14.0
3 months.....	11.7	16.3
6 months.....	14.0	16.4
9 months.....	10.8	12.6
12 months.....	10.8	12.2

### PART III—MICROANALYSES

At the end of the drying period of the 1961 study, many dead insects were observed among the raisins dried on malathion-treated trays (fig. 5). Insects were attracted to the drying grapes on the treated trays and were killed by the malathion. Relatively few dead insects were found among raisins dried on untreated trays, since the insects attracted to these trays migrated before the end of the drying period. Similar studies in 1962 revealed the same trend, except that the insect population was less than in the previous year. Since the treated trays did have dead insects, it was decided early in the study to include data on how the presence of dead insects on the trays affected the amount of insect fragments on the finished raisins, as determined by microanalysis.

This part of the report contains data on microanalyses of the raisins before and after processing. Since this information has not previously been published for the 1961–62 studies, it is given here. The data from microanalyses of raisins in the 1962–63 studies are also given.

#### Materials and Methods

In microanalysis, samples of about 4 ounces of natural raisins or 8 ounces of processed raisins were placed in 2-liter Erlenmeyer flasks, and one-sixth teaspoon of household detergent was added to each flask. Sufficient tapwater was added to raise the water level  $2\frac{1}{2}$  to 3 inches in the flasks. The raisins were permitted to soak or rehydrate in the detergent solution overnight. When the raisins resembled the shape of grapes,

each flask was agitated for 5 minutes. The rehydrated raisins in each flask were washed thoroughly and all of the water used in soaking and rinsing was passed through 7-cm. ruled filter papers to collect all foreign material removed from the raisins. The papers were examined under 20 to  $30\times$  magnification, and the amount and kind of foreign material found was recorded (7, Sec. 3.1).

#### 1961–62 Studies

The grapes used in these studies were produced and dried into raisins in a vineyard that had a history of moderate to severe bunch rot contamination, and as a result large numbers of drosophila, dried fruit beetles, and raisin moths were attracted to the area. This was particularly true of these grapes because they were removed from the vines and placed on trays to dry late in the season at a time when the incidence of bunch rot and the number of insects were greatest. Yerington and Spitler (8) found that injured grapes also attracted insects when placed on trays to dry. Careful picking gives better raisin quality. The procedures used in dusting ripening grapes on the vines, drying the grapes into raisins on malathion-treated trays, storing the raisins, sampling, and screening and processing the raisins and data on effectiveness of malathion were described by Nelson and others (2). Their publication did not include data on the insect fragments in the raisins.

Analyses to determine the number of insects and insect fragments in the raisins were made by the U.S. Department of Agriculture. Three replicate samples of raisins were taken from stacks that were dried on each combination of trays and treatments. Four-ounce subsamples of natural raisins and eight-ounce subsamples of processed raisins were drawn from the three replicate samples. These subsamples were taken from the processed raisins at the time they were processed on October 31, 1961, which was also the date that the raisins were placed in storage. Samples were also taken from the natural and processed raisins at the end of 5 months and at the end of 1 year in storage.

#### 1962–63 Studies

The procedures for grape dusting, tray treatment, and storing and processing of the raisins used in studies conducted on the 1962 crop are given in Part I of this publication. Samples of raisins for microanalyses were taken in the same manner as those in the 1961–62 studies, except that each sample was split into two parts. One part was examined by the U.S. Department of



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FIGURE 5.—Dead insects among raisins dried on malathion-treated paper trays.

Agriculture and the other by the Dried Fruit Association of California. The same procedure was followed by both groups in conducting the analyses.

## Results

### 1961-62 Studies

The results of the microanalyses are presented in tables 8 and 9. In general, there were more insects and insect fragments found on the raisins dried on treated trays than on those dried on untreated trays.

In the studies reported here, insects among the raisins on the trays were not removed at the time the raisins were boxed and stored. If the raisins had been put over a raisin cleaner at the time of storage, the number of insects, parts, eggs, and fragments would have been considerably reduced in the natural as well as in the processed raisins.

During the first 5 months in storage a small hole in the center of the stack permitted moisture to enter, and some of the raisins became moist. The raisins located just beneath the opening in the roof happened to be those that

were dried on the untreated trays and were affected most. This moist condition of the raisins stimulated development of large numbers of drosophila. Many were killed when they entered boxes containing raisins that had been dried on treated trays. Even though there were large numbers of insects and insect fragments on the natural raisins, there were fewer insects and insect fragments on the raisins made from dusted grapes. Comparative samples of processed raisins were taken at the beginning, and samples of processed raisins were taken at the end of 5 months and 12 months in storage. The data presented in tables 8 and 9 show that processing reduced the number of insects and insect parts on the raisins.

### 1962-63 Studies

The numbers of insects and insect parts present on samples of natural and processed raisins as determined by microanalysis are summarized in tables 10, 11, 12, and 13. These are the averaged results obtained by the U.S. Department of Agriculture and the Dried Fruit Association of California. In general, the natural raisins dried in vineyard No. 2 contained more insects and

TABLE 8.—*Summary of microanalysis counts of insects and insect fragments found on a 4-oz. subsample of natural raisins grouped according to length of storage and grape and tray treatments, 1961-62*

Origin of raisins and length of storage	Malathion residue <sup>1</sup> on trays	Drosophila <sup>2</sup>	Dried-fruit beetle <sup>3</sup>	Saw-toothed grain beetle <sup>3</sup>	Moth <sup>3</sup>	Other insects <sup>2</sup>	Total insects	Insect fragments	Total
Undusted grapes	Mg./sq. ft.	Number	Number	Number	Number	Number	Number	Number	Number
5 months....	< 0.5	125	3	2	6	25	161	224	385
12 months....	< 0.5	179	15	3	72	100	369	1,123	1,492
Total.....		304	18	5	78	125	530	1,347	1,877
5 months....	428.4	123	0	0	1	7	131	188	319
12 months....	428.4	1,020	165	3	172	1,132	2,492	1,400	3,892
Total.....		1,143	165	3	173	1,139	2,623	1,588	4,211
5 months....	806.4	745	3	0	1	28	777	496	1,273
12 months....	806.4	1,238	16	0	45	102	1,401	779	2,180
Total.....		1,983	19	0	46	130	2,178	1,275	3,453
Dusted grapes									
5 months....	< 0.5	109	8	0	7	21	145	16	161
12 months....	< 0.5	297	36	0	63	186	582	1,629	2,211
Total.....		406	44	0	70	207	727	1,645	2,372
5 months....	428.4	910	8	0	2	27	947	439	1,386
12 months....	428.4	351	6	0	12	42	411	603	1,014
Total.....		1,261	14	0	14	69	1,358	1,042	2,400
5 months....	806.4	311	11	0	0	8	330	220	550
12 months....	806.4	360	0	0	105	81	546	201	747
Total.....		671	11	0	105	89	876	421	1,297

<sup>1</sup>Sensitivity of chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Eggs, larvae, pupae, and adults.

<sup>3</sup>Larvae, pupae, and adults.

TABLE 9.—Summary of microanalysis counts of the number of insects and insect fragments found on an 8-oz. subsample of processed raisins grouped according to length of storage and grape and tray treatments, 1961–62

Origin of raisins and length of storage	Malathion residue <sup>1</sup> on trays	Drosophila <sup>2</sup>	Dried-fruit beetle <sup>3</sup>	Saw-toothed grain beetle <sup>3</sup>	Moth <sup>3</sup>	Other insects <sup>2</sup>	Total insects	Insect fragments	Total
<b>Undusted grapes</b>	<i>Mg./sq. ft.</i>	Number	Number	Number	Number	Number	Number	Number	Number
5 months.....	< 0.5	39	0	0	1	5	45	44	89
12 months.....	< 0.5	18	2	0	4	17	41	133	174
Total.....		57	2	0	5	22	86	177	263
5 months.....	428.4	34	9	0	0	1	44	52	96
12 months.....	428.4	38	13	0	0	22	73	74	147
Total.....		72	22	0	0	23	117	126	243
5 months.....	806.4	104	5	0	0	3	112	148	260
12 months.....	806.4	98	3	0	0	10	111	51	162
Total.....		202	8	0	0	13	223	199	422
<b>Dusted grapes</b>									
5 months.....	< 0.5	12	1	0	1	2	16	20	36
12 months.....	< 0.5	54	4	0	0	9	67	118	185
Total.....		66	5	0	1	11	83	138	221
5 months.....	428.4	63	3	0	0	8	74	157	231
12 months.....	428.4	48	2	0	0	4	54	60	114
Total.....		111	5	0	0	12	128	217	345
5 months.....	806.4	33	0	3	0	4	40	67	107
12 months.....	806.4	35	0	0	0	12	47	46	93
Total.....		68	0	3	0	16	87	113	200

<sup>1</sup>Sensitivity of chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Eggs, larvae, pupae, and adults.

<sup>3</sup>Larvae, pupae, and adults.

insect parts than those dried in vineyard No. 1. The raisins dried on untreated trays in vineyard No. 2 contained fewer insects and insect fragments when placed in storage than those dried on treated trays. Processing removed about 90 percent of the insect material present on the natural raisins. Processed raisins from each vineyard contained similar amounts of insects and insect fragments.

## Discussion

These studies show a marked variation in the insect population on the trays, depending on the location of the vineyard and the amount of bunch rot and mechanically injured grapes present, as well as on the particular season and year. Insects attracted to the raisins being dried on the malathion-treated trays were killed by the malathion.

Studies conducted by Simmons and others (3,4,5), Donohoe and others (1), and the Stored-Product Insects Section (6) had previously demonstrated that screening with a raisin cleaner (fig. 6) removed a large percentage of the dead and live insects, eggs, and larvae present in natural raisins at the time of boxing and storage.



FIGURE 6.—Cleaner used for removing foreign material from natural raisins.

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TABLE 10.—Summary of microanalysis counts of the average number of insects and insect fragments found on two 4-oz. subsamples of natural raisins dried on untreated and malathion-treated trays in vineyard No. 1, by tray treatment and length of storage, 1962-63

Malathion residue <sup>1</sup> on trays and storage period	Drosophila <sup>2</sup>	Dried- fruit beetle <sup>3</sup>	Saw-toothed grain beetle <sup>3</sup>	Moth <sup>3</sup>	Other insects <sup>2</sup>	Total insects	Insect fragments	Total
	Number	Number	Number	Number	Number	Number	Number	Number
0.5 mg./sq. ft.:								
None.....	0	0	0	24	54	78	72	150
3 months.....	1	0	0	3	6	10	49	59
6 months.....	4	0	0	0	23	27	62	89
9 months.....	0	1	0	0	5	6	45	51
12 months.....	14	6	10	53	7	90	114	204
Total.....	19	7	10	80	95	211	342	553
31.2 mg. sq. ft.:								
None.....	1	0	0	3	37	41	78	119
3 months.....	2	1	0	0	9	12	44	56
6 months.....	0	1	0	0	38	39	53	92
9 months.....	0	2	0	0	11	13	29	42
12 months.....	2	1	0	5	4	12	54	66
Total.....	5	5	0	8	99	117	258	375
152.4 mg./sq. ft.:								
None.....	0	3	0	2	28	33	46	79
3 months.....	3	0	0	2	5	10	29	39
6 months.....	86	3	0	0	13	102	47	149
9 months.....	0	0	0	2	9	11	27	38
12 months.....	0	1	3	5	12	21	52	73
Total.....	89	7	3	11	67	177	201	378
304.2 mg./sq. ft.:								
None.....	0	2	0	1	45	48	83	131
3 months.....	0	0	0	0	15	15	37	52
6 months.....	0	1	0	0	34	35	40	75
9 months.....	0	0	2	1	4	7	19	26
12 months.....	2	6	0	1	17	26	43	69
Total.....	2	9	2	3	115	131	222	353

<sup>1</sup>Sensitivity of chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Eggs, larvae, pupae, and adults.

<sup>3</sup>Larvae, pupae, and adults.

TABLE 11.—Summary of microanalysis counts of the average number of insects and insect fragments found on two 8-oz. subsamples of processed raisins dried on untreated and malathion-treated trays in vineyard No. 1, by tray treatment and length of storage, 1962-63

Malathion residue <sup>1</sup> on trays and storage period	Drosophila <sup>2</sup>	Dried- fruit beetle <sup>3</sup>	Saw-toothed grain beetle <sup>3</sup>	Moth <sup>3</sup>	Other insects <sup>2</sup>	Total insects	Insect fragments	Total
	Number	Number	Number	Number	Number	Number	Number	Number
0.5 mg., sq. ft.:								
None.....	28	0	0	0	12	40	17	57
3 months.....	0	0	0	0	1	1	8	9
6 months.....	2	0	0	0	0	2	7	9
9 months.....	0	0	0	0	0	0	3	3
12 months.....	3	0	0	1	0	4	7	11
Total.....	33	0	0	1	13	47	42	89
31.2 mg./sq. ft.:								
None.....	0	0	0	0	1	1	1	2
3 months.....	0	0	0	0	3	3	4	7
6 months.....	0	0	0	0	0	0	5	5
9 months.....	0	0	0	0	3	3	10	13
12 months.....	0	0	0	0	0	0	9	9
Total.....	0	0	0	0	7	7	29	36

TABLE 11. — Summary of microanalysis counts of the average number of insects and insect fragments found on two 8-oz. subsamples of processed raisins dried on untreated and malathion-treated trays in vineyard No. 1, by tray treatment and length of storage, 1962-63 — Continued

Malathion residue <sup>1</sup> on trays and storage period	Drosophila <sup>2</sup>	Dried- fruit beetle <sup>3</sup>	Saw-toothed grain beetle <sup>3</sup>	Moth <sup>3</sup>	Other insects <sup>2</sup>	Total insects	Insect fragments	Total
	Number	Number	Number	Number	Number	Number	Number	Number
152.4 mg./sq. ft.:								
None.....	0	0	0	0	2	2	2	4
3 months.....	1	0	0	0	0	1	5	6
6 months.....	1	0	0	0	0	1	0	1
9 months.....	0	1	0	0	0	1	0	1
12 months.....	0	1	0	0	0	1	8	9
Total.....	2	2	0	0	2	6	15	21
304.2 mg./sq. ft.:								
None.....	0	0	0	0	0	0	0	0
3 months.....	0	0	0	0	1	1	5	6
6 months.....	0	0	0	0	1	1	5	6
9 months.....	0	0	0	0	2	2	2	4
12 months.....	0	0	1	0	0	1	14	15
Total.....	0	0	1	0	4	5	26	31

<sup>1</sup>Sensitivity of chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Eggs, larvae, pupae, and adults.

<sup>3</sup>Larvae, pupae, and adults.

TABLE 12. — Summary of microanalysis counts for the average number of insects and insect fragments found in two 4-oz. subsamples of natural raisins dried on untreated and malathion-treated trays in vineyard No. 2, by tray treatment and length of storage, 1962-63

Malathion residue <sup>1</sup> on trays and storage period	Drosophila <sup>2</sup>	Dried- fruit beetle <sup>3</sup>	Saw-toothed grain beetle <sup>3</sup>	Moth <sup>3</sup>	Other insects <sup>2</sup>	Total insects	Insect fragments	Total
	Number	Number	Number	Number	Number	Number	Number	Number
< 0.5 mg./sq. ft.:								
None.....	1	0	0	29	60	90	97	187
3 months.....	6	0	0	1	7	14	45	59
6 months.....	1	0	0	0	34	35	72	107
9 months.....	1	4	0	3	30	38	69	107
12 months.....	1	1	0	32	15	49	228	277
Total.....	10	5	0	65	146	226	511	737
34.2 mg./sq. ft.:								
None.....	3	3	0	3	108	117	226	343
3 months.....	3	0	0	0	23	26	49	75
6 months.....	213	0	0	1	42	256	53	309
9 months.....	1	2	1	1	13	18	28	46
12 months.....	0	6	0	7	24	37	35	72
Total.....	220	11	1	12	210	454	391	845
150.2 mg./sq. ft.:								
None.....	12	4	0	0	79	95	150	245
3 months.....	1	1	0	2	10	14	35	49
6 months.....	3	0	1	0	20	24	52	76
9 months.....	0	1	0	1	16	18	34	52
12 months.....	1	4	1	1	5	12	17	29
Total.....	17	10	2	4	130	163	288	451
290.4 mg./sq. ft.:								
None.....	0	4	0	4	94	102	183	285
3 months.....	1	0	0	0	9	10	14	24
6 months.....	5	0	0	0	23	28	47	75
9 months.....	0	2	1	0	12	15	23	38
12 months.....	3	6	1	0	13	23	25	48
Total.....	9	12	2	4	151	178	292	470

<sup>1</sup>Sensitivity of chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Eggs, larvae, pupae, and adults.

<sup>3</sup>Larvae, pupae, and adults.

TABLE 13.—Summary of microanalysis counts of the average number of insects and insect fragments found in two 8-oz. subsamples of processed raisins dried on untreated and malathion-treated trays in vineyard No. 2, by tray treatment and length of storage, 1962-63

Malathion residue <sup>1</sup> on trays and storage period	Drosophila <sup>2</sup>	Dried- fruit beetle <sup>3</sup>	Saw-toothed grain beetle <sup>3</sup>	Moth <sup>3</sup>	Other insects <sup>2</sup>	Total insects	Insect fragments	Total
<0.5 mg./sq. ft.:	Number	Number	Number	Number	Number	Number	Number	Number
None.....	0	0	0	0	1	1	4	5
3 months.....	0	0	0	1	0	1	4	5
6 months.....	1	0	0	1	3	5	7	12
9 months.....	0	0	0	1	1	2	12	14
12 months.....	0	1	0	1	0	2	22	24
Total.....	1	1	0	4	5	11	49	60
34.2 mg./sq. ft.:	Number	Number	Number	Number	Number	Number	Number	Number
None.....	5	0	0	0	1	6	5	11
3 months.....	1	0	0	0	0	1	4	5
6 months.....	1	0	0	0	1	2	9	11
9 months.....	0	0	1	0	0	1	2	3
12 months.....	0	0	0	3	3	6	5	11
Total.....	7	0	1	3	5	16	25	41
150.2 mg./sq. ft.:	Number	Number	Number	Number	Number	Number	Number	Number
None.....	1	0	0	0	4	5	2	7
3 months.....	0	0	0	0	6	6	6	12
6 months.....	1	0	0	0	0	1	18	19
9 months.....	0	1	2	0	0	3	3	6
12 months.....	0	0	2	0	1	3	16	19
Total.....	2	1	4	0	11	18	45	63
290.4 mg./sq. ft.:	Number	Number	Number	Number	Number	Number	Number	Number
None.....	0	0	0	0	2	2	1	3
3 months.....	1	0	0	0	1	2	1	3
6 months.....	1	0	0	0	0	1	0	1
9 months.....	0	2	0	1	2	5	5	10
12 months.....	0	1	0	0	1	2	18	20
Total.....	2	3	0	1	6	12	25	37

<sup>1</sup>Sensitivity of chemical analytical method is 0.5 p.p.m.

<sup>2</sup>Eggs, larvae, pupae, and adults.

<sup>3</sup>Larvae, pupae, and adults.

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